

Calibration-Round-Robin CaRo 18

Final Report

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1. Conclusion

According to international standards, test equipment must be calibrated at intervals by comparison with a standard or a calibrated testing apparatus. This calibration also applies to the 20-l-apparatus and the 1-m³-vessel for the determination of Pmax and Kmax and the apparatus for determination of the minimum ignition energy. The test procedure is an important part of this calibration. A general check at the component level is incomplete and hence inadmissible.

Unfortunately there are neither internationally recognized reference samples nor reference equipment available for the determination of these explosion characteristics. Therefore the following calibration method has been carried out successfully:

A dust has been selected, prepared and supplied to **21** test laboratories all over the world. The mean values of the explosion indices, measured by the participating laboratories, has been calculated as reference values. The testing laboratories have been informed about the evaluation with a certificate.

This report presents the results of this calibration method and describes the evaluation procedures.

CaRo 18 – Reference values for the Explosion Indices Pmax and Kmax

Pmax (bar)	8.4 ± 10% (7.6 ... 9.2)
Kmax (bar·m/s)	252 ± 10% (227 ... 277)

CaRo 18 – Reference values for the Minimum Ignition Energy MIE

Es / 3	Es	Es · 3
0.6 mJ	1.8 mJ	5.3 mJ

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1.1 Participants:

Further details about participants who have agreed to a publication, can be found in the final section.

	Pmax, Kmax (19)		MIE (22)	
	20-l	1 m ³	MIKE	others
Australia	1		1	
Brazil	1		1	
Canada	1			1
China	2		1	
Germany	1		4	
Italy	1		1	
Japan			1	
Switzerland	4		4	
United Kingdom	5		3	2
USA	3		3	
Total:	19		19	3

1.2 Test substance:

For correct calibration the CaRo 18 test sample has been milled, homogenized and shipped in an air tight package.

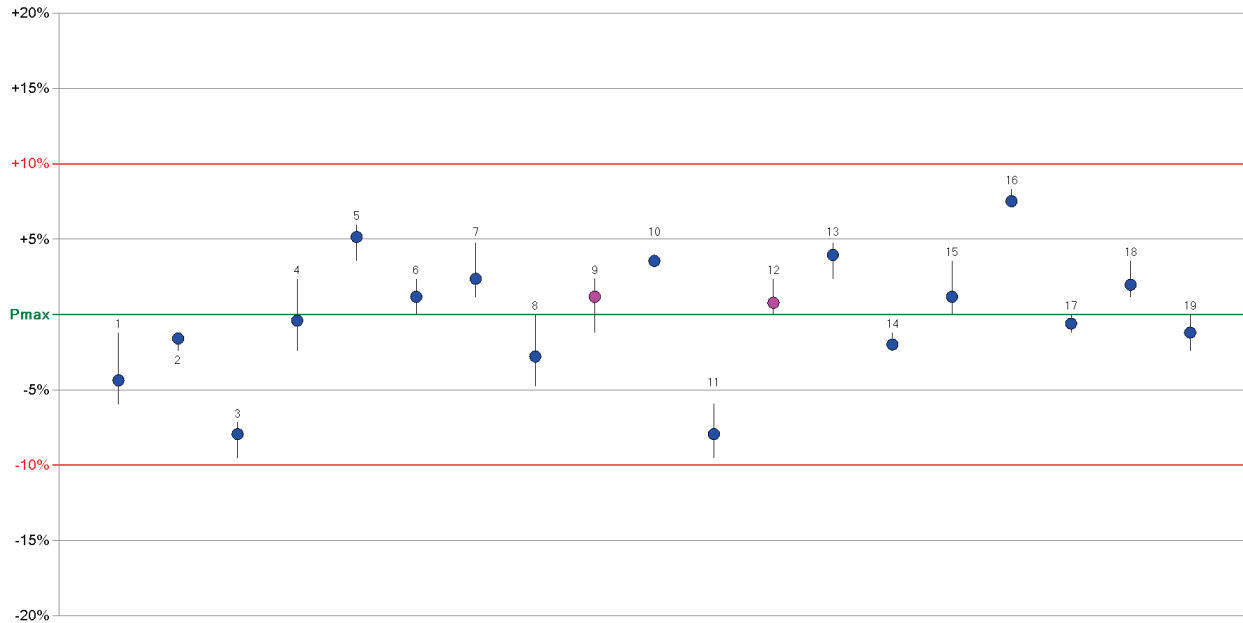
Therefore the sample has to be tested „as delivered“.

CaRo 18 = Niacin CaRo Test Dust (Nicotinic acid)

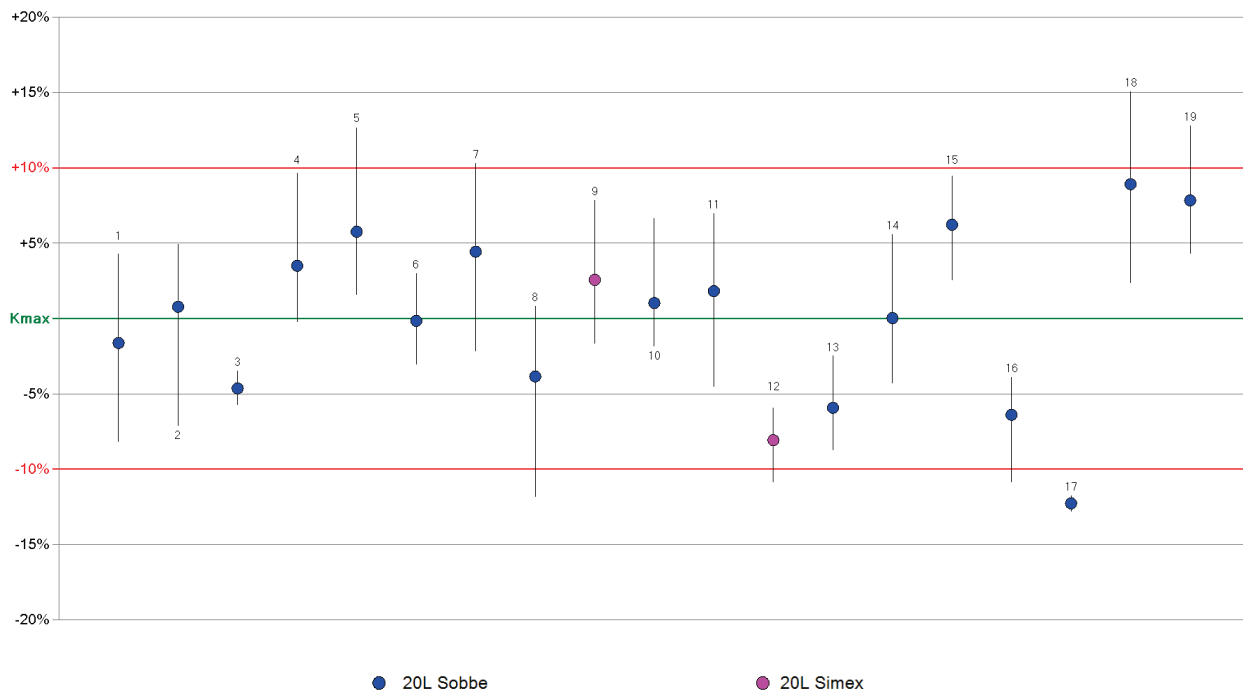
Particle size:	d 10 [µm]	d 50 = median [µm]	d 90 [µm]
Sample 1	3.1	20.1	73.2
Sample 2	3.1	19.6	74.7
Sample 3	3.2	21.2	79.4
Sample 4	3.0	20.5	73.9

2. Explosion Indices Pmax, Kmax

Pmax = 8.4 bar ± 10% (7.6 ... 9.2) at 509 g/m³



Kmax = 252 bar·m/s ± 10% (227 ... 277) at 632 g/m³



The individual results are drawn in relation to the arithmetic mean of all results and in chronological sequence (number of certificate).

2.1 Test procedure:

The method for determination of P_{max} , K_{max} is described in the „Manual CaRo 18“

2.2 Evaluation:

The explosion indices P_{max} and $(dP/dt)_{max}$ are defined as the mean values of the maximum values of each series. Subsequently, the explosion index K_{max} is calculated from the mean value $(dP/dt)_{max}$.

2.3 Scatter of P_{max} and K_{max} :

The maxima of each series must not deviate by more than **10%** of P_{max} resp. K_{max} .
Otherwise this series must be repeated!

2.4 Calculation of the reference values:

First the mean values of all test results has been calculated. In a second step all results outside of the tolerance band are excluded prior to the subsequent calculation of the mean value. Due to the large number of participants the mean values did not change.

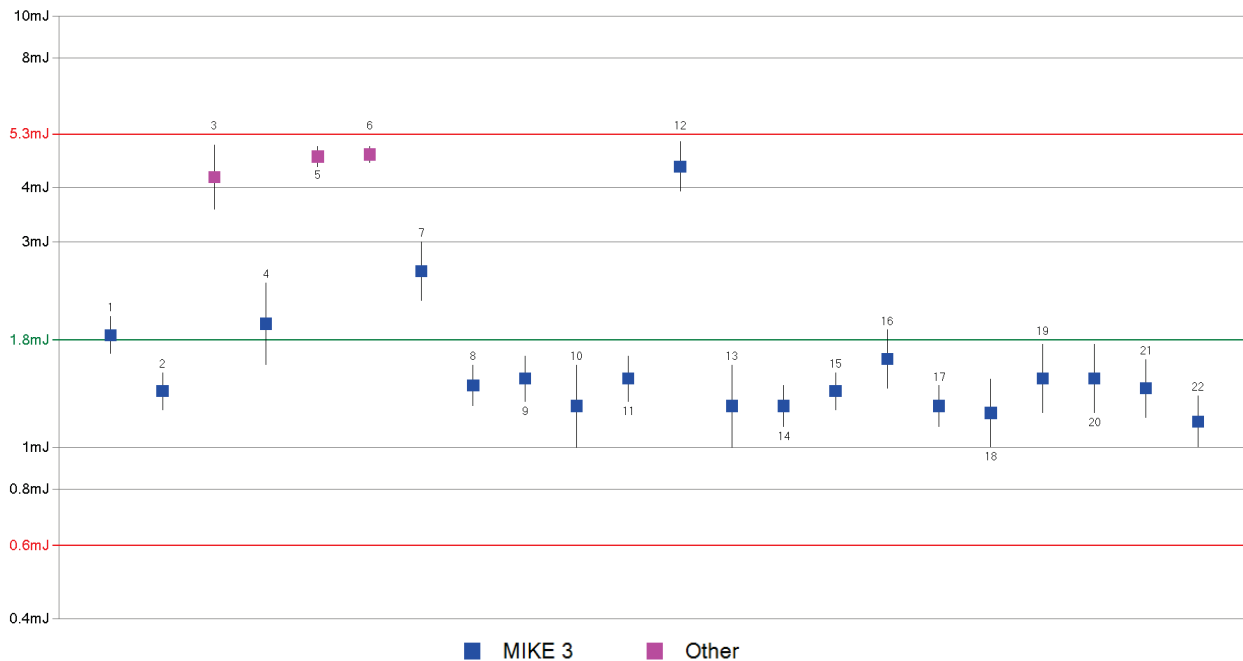
2.5 Cause of errors:

Some laboratories had to repeat the tests.

The reasons were:

- a. Use of synthetic air instead of normal compressor compressed air.
Synthetic compressed air can result in lower values for the explosion indices P_{max} and K_{max} and higher values for the MIE.
We highly recommend to refrain from using synthetic compressed air.
- b. Too high or too low initial pressure P_i .
The explosion indices P_{max} and K_{max} are direct proportional to the initial pressure P_i , the pressure in the sphere at the moment of ignition.
This relation is linear up to an initial pressure of approx. 3 bar.
- c. Twisted or misplaced cabling between pressure sensors and charge amplifiers resulting in strongly deviating pressure curves.

3. Minimum Ignition Energy MIE



The individual results are drawn in chronological sequence (number of certificate).

3.1 Test procedure:

The method for determination of the MIE is described in the „Manual CaRo 18“.

3.2 Estimation of the statistical energy (Es):

The minimum ignition energy MIE lies, by definition, between two energy values: $E_1 < MIE < E_2$

For the purpose of comparison between different apparatus, only one MIE value (Es) instead of the energy range (E1, E2) shall be used. This single value (Es) can be estimated by use of the probability of ignition as follows (EN 13821):

Provided that for the energy E2 a minimum of 5 dust concentrations evenly distributed are tested, the position of the MIE in the E1-E2 range can be estimated. At ignition energy E2, the number of dust concentrations with ignition, is divided by the total number of dust concentrations tested.

$$E_S = 10^{\frac{\log E_2 - \frac{I[E_2] \cdot (\log E_2 - \log E_1)}{(NI + I) \cdot [E_2] + 1}}{10}}$$

where is:
 $I[E_2]$ = number of tests with ignition at energy E2
 $(NI+I) [E_2]$ = total number of tests at energy E2

3.3 Criteria for conformity:

Conformity in the CaRo 18 is given, when the Es-value of each equipment differ less than a factor of 3 to the mean (Es) of all equipment:

Es / 3	Es	Es • 3
0.6 mJ	1.8 mJ	5.3 mJ

3.4 Cause of errors:

The use of synthetic compressed resulted in a change in the ignition behavior.

4. List of Participants

Country	Company Laboratory	E-Mail	Pmax Kmax	MIE
Australia	Simtars – Department of Natural Resources, Mines and Energy	negar.fasihiani@simtars.com.au	✓	✓
Brazil	IPT – Instituto de Pesquisas Tecnológicas do Est. de São Paulo	ricalca@ipt.br	✓	✓
Canada	Jensen Hughes	mclouthier@jensenhughes.com	✓	✓
China	Dekra Testing and Certification (Shanghai)	dengping.hu@dekra.com	✓	
Germany	BASF SE	johannes.a.fischer@basf.com	✓	✓
Germany	Merck KGaA	thomas.keil@merckgroup.com		✓
Germany	Sanofi-Aventis Deutschland GmbH	dirk.hoerstermann@sanofi.com		✓
Italy	Innovhub – SSI S.r.l.	antonella.mazzei@mi.camcom.it	✓	✓
Japan	Technology Institution of Industrial Safety	nishimura@tiis.or.jp		✓
Switzerland	DSM	romeo.isner@dsm.com		✓
Switzerland	Givaudan International SA	chantal.berchten@givaudan.com	✓	✓
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Switzerland	TÜV SÜD Process Safety	mischa.schwaninger@tuev-sued.ch	✓	✓
United Kingdom	BRE Global	lee.amendt@bre.co.uk	✓	✓
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